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प्लास्टिक — परीक्षण पद्धतियाँ

भाग 8 स्थायी/रासायनिक गुणधर्म

अनुभाग 11 पर्यावरणात्मक प्रतिबल-भंजन (ई एस सी) ज्ञात करना — सतत-तनन प्रतिबल पद्धति

Indian Standard

PLASTICS — METHODS OF TESTING

PART 8 PERMANENCE/CHEMICAL PROPERTIES

Section 11 Determination of Environmental Stress Cracking (ESC) — Constant-Tensile Stress Method

ICS 83.080; 19.060

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NEW DELHI 110002

April 1999

Price Group 4

Methods of Test for Plastics Sectional Committee, PCD 23

NATIONAL FOREWORD

This Indian Standard (Part 8/Sec 11) which is identical with ISO 6252 : 1992 'Plastics — Determination of environmental stress cracking (ESC) — Constant-tensile-stress method' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of Methods of Test for Plastics Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 150 : 1980 Raw, refined and boiled linseed oil for paints and varnishes — Specification and methods of test	Nil	—
ISO 291 : 1997 Plastics — Standard atmospheres for conditioning and testing	IS 196 : 1966 Atmospheric conditions for testing (*revised*)	Technically equivalent with minor deviations
ISO 293 : 1986 Plastics — Compression moulding test specimens of thermoplastic materials	IS 13360 (Part 2/Sec 1) : 1992 Plastics — Methods of testing : Part 2 Sampling and preparation of test specimens, Section 1 Compression moulding test specimens of thermoplastic materials	Technically equivalent
ISO 294 : 1995 Plastics — Injection moulding of test specimens of thermoplastic materials	IS 8543 (Part 3/Sec 2) : 1978 Methods of testing plastics : Part 3 Preparation of test specimens, Section 2 Injection moulded test specimens	Not technically equivalent in all respects. However, this Indian Standard will be considered for revision to align it with ISO as and when the revised version of ISO 294 : 1995 is available
ISO 527 : 1966 Plastics — Determination of tensile properties	IS 8543 (Part 4/Sec 1) : 1984 Plastics — Methods of testing : Part 4 Short term machanical properties, Section 1 Determination of tensile properties	Technically equivalent

(*Continued on third cover*)

Indian Standard

PLASTICS — METHODS OF TESTING

PART 8 PERMANENCE/CHEMICAL PROPERTIES

Section 11 Determination of Environmental Stress Cracking (ESC) — Constant-Tensile Stress Method

1 Scope

This International Standard specifies methods for the determination of environmental stress cracking (ESC) of plastics when they are subjected to a constant tensile force in the presence of chemical agents.

It is applicable to test specimens prepared by moulding and/or machining and can be used both for the assessment of ESC of plastics materials exposed to different environments, and for the determination of ESC of different plastics materials exposed to a specific environment.

NOTE 1 Methods for the determination of environmental stress cracking by means of a constant-strain test are specified in ISO 4599 and ISO 4600.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 150:1980, *Raw, refined and boiled linseed oil for paints and varnishes — Specifications and methods of test.*

ISO 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 294:1975, *Plastics — Injection moulding test specimens of thermoplastic materials.*

ISO/R 527:1966, *Plastics — Determination of tensile properties.*

ISO 899:1981, *Plastics — Determination of tensile creep.*

ISO 2557-1:1989, *Plastics — Amorphous thermoplastics — Preparation of test specimens with a specified maximum reversion — Part 1: Bars.*

ISO 2818:1980, *Plastics — Preparation of test specimens by machining.*

ISO 3167:1983, *Plastics — Preparation and use of multipurpose test specimens.*

ISO 4599:1986, *Plastics — Determination of resistance to environmental stress cracking (ESC) — Bent strip method.*

ISO 4600:1992, *Plastics — Determination of environmental stress cracking (ESC) — Ball or pin impression method.*

3 Principle

A test specimen is subjected to a constant tensile force, corresponding to a stress lower than that at yield, while immersed in a specified environment at the temperature selected for testing. The time and/or stress at which the specimen breaks is recorded.

The environmental stress cracking of the test specimens is determined by one of the following methods (A, B or C), depending upon the time to rupture:

- Method A: Determination of the tensile stress leading to rupture at 100 h. This stress is obtained by interpolation of the graph of time to rupture versus applied tensile stress.
- Method B: Determination of the time to rupture under a specified tensile stress. This method is used when the time to rupture exceeds 100 h.

— Method C: Determination of the tensile stress versus time to rupture curve up to an agreed time.

NOTE 2 If creep measurements are desired, the method outlined in ISO 899 is followed.

4 Apparatus

4.1 Testing device, allowing test specimens to be submitted simultaneously to a tensile stress and to the chemical environment.

If the chemical is a liquid at the test temperature, the test specimen shall be completely immersed in it. If it is highly viscous at the test temperature, the specimens may be covered with a coating of the agent at least 2 mm thick (see clause 5).

The parts of the device that come into contact with the chemical shall be made of an inert material, for example stainless steel.

The constant tensile stress may be applied with weights (figures 1 and 2 are schematic diagrams of suitable apparatus). The force shall be accurate to ± 1 %. Care shall be taken to ensure that there is no loss of stress in the system, for example by friction.

If the testing device has several test stations, means shall be provided to prevent the vibration occurring through failure at one station from being transmitted to the whole system.

Care shall be taken that the specimens are subjected only to forces parallel to their longitudinal axis, and not to bending or twisting forces.

4.2 Temperature-controlled bath or room, allowing the test system to be maintained at (23 ± 1) °C or at a higher test temperature up to 105 °C to within ± 0,5 °C (see clause 6).

4.3 Automatic timer, as shown schematically in figure 2, to measure the time to rupture of each specimen to 1 % or better.

4.4 Equipment for the preparation of test specimens by moulding (see ISO 293 and ISO 294), machining (see ISO 2818) or die cutting.

5 Chemical environment

The chemical environment used for the test shall be that specified in the relevant International Standard for the material tested. If nothing is specified, use either the agent which will be in contact with the material in the expected application or a reference product agreed upon between the interested parties.

NOTE 3 Examples of reference products are:

a) 95 % (*V*/*V*) ethanol — pharmaceutical quality;

b) a 1 % (*m*/*m*) solution of nonylphenoxy-poly(ethyleneoxy)ethanol[1] in distilled water;

c) refined linseed oil (see ISO 150).

6 Conditioning and test conditions

6.1 Conditioning

Unless otherwise agreed between the interested parties, the test specimens shall be conditioned before testing for at least 24 h at (23 ± 2) °C and (50 ± 5) % relative humidity (see ISO 291).

6.2 Test temperature

The preferred test temperatures are (23 ± 1) °C and (55 ± 0,5) °C. If required, other temperatures may be used, preferably selected from the following:

(40 ± 0,5) °C

(70 ± 0,5) °C

(85 ± 0,5) °C

(100 ± 0,5) °C

or as agreed upon by the interested parties.

7 Test stress

7.1 Maximum permissible stress

The stress applied to the test specimen during the test shall be less than the tensile stress at yield of the material at the temperature of the test.

NOTE 4 As a general guide, the stress that produces an elongation of 2 % after 1 h can be taken as the maximum permissible stress. This stress can be determined by preliminary tests using several different stresses.

7.2 Method A

Determine the tensile stress required to cause rupture after 100 h by applying a series of stresses, the maximum stress being as defined in 7.1.

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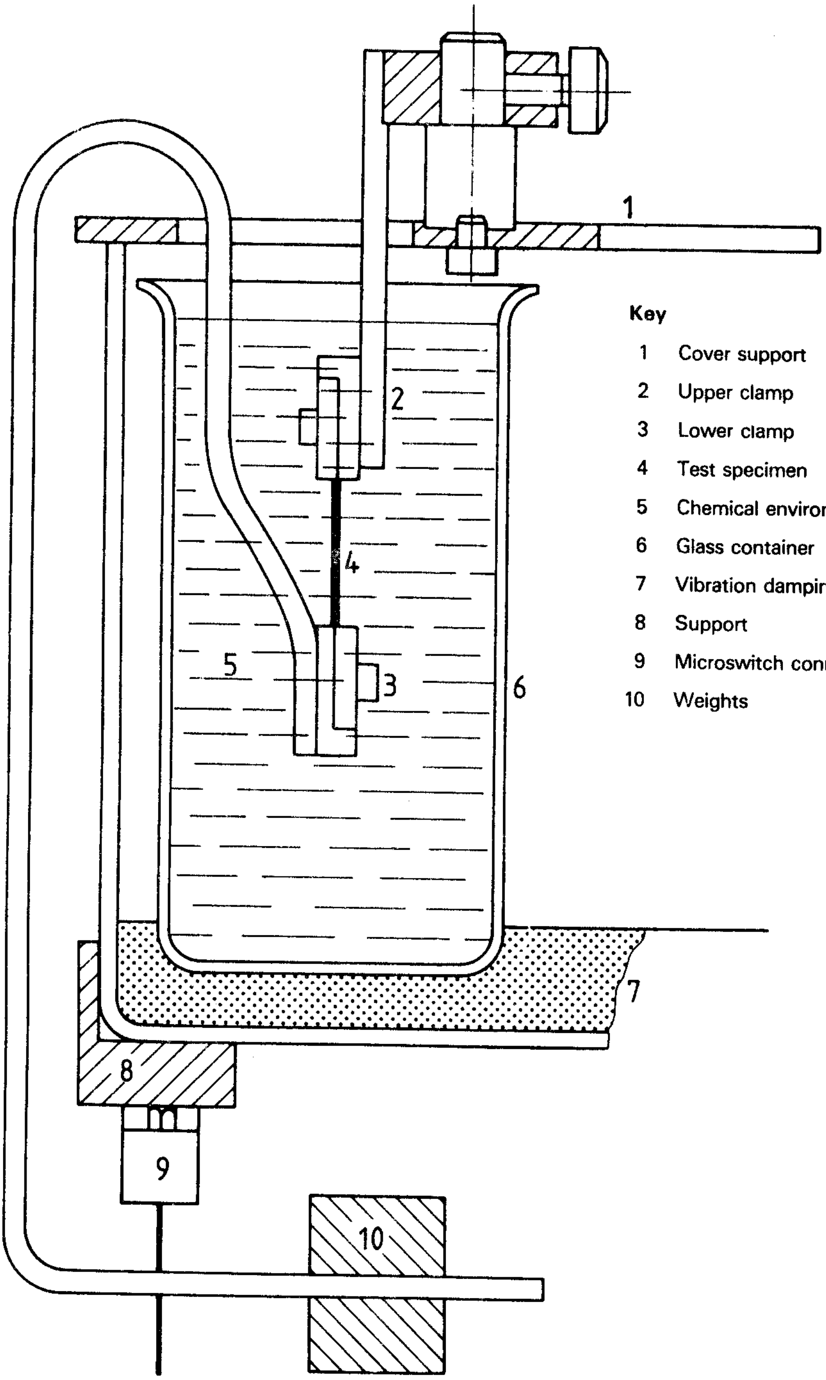
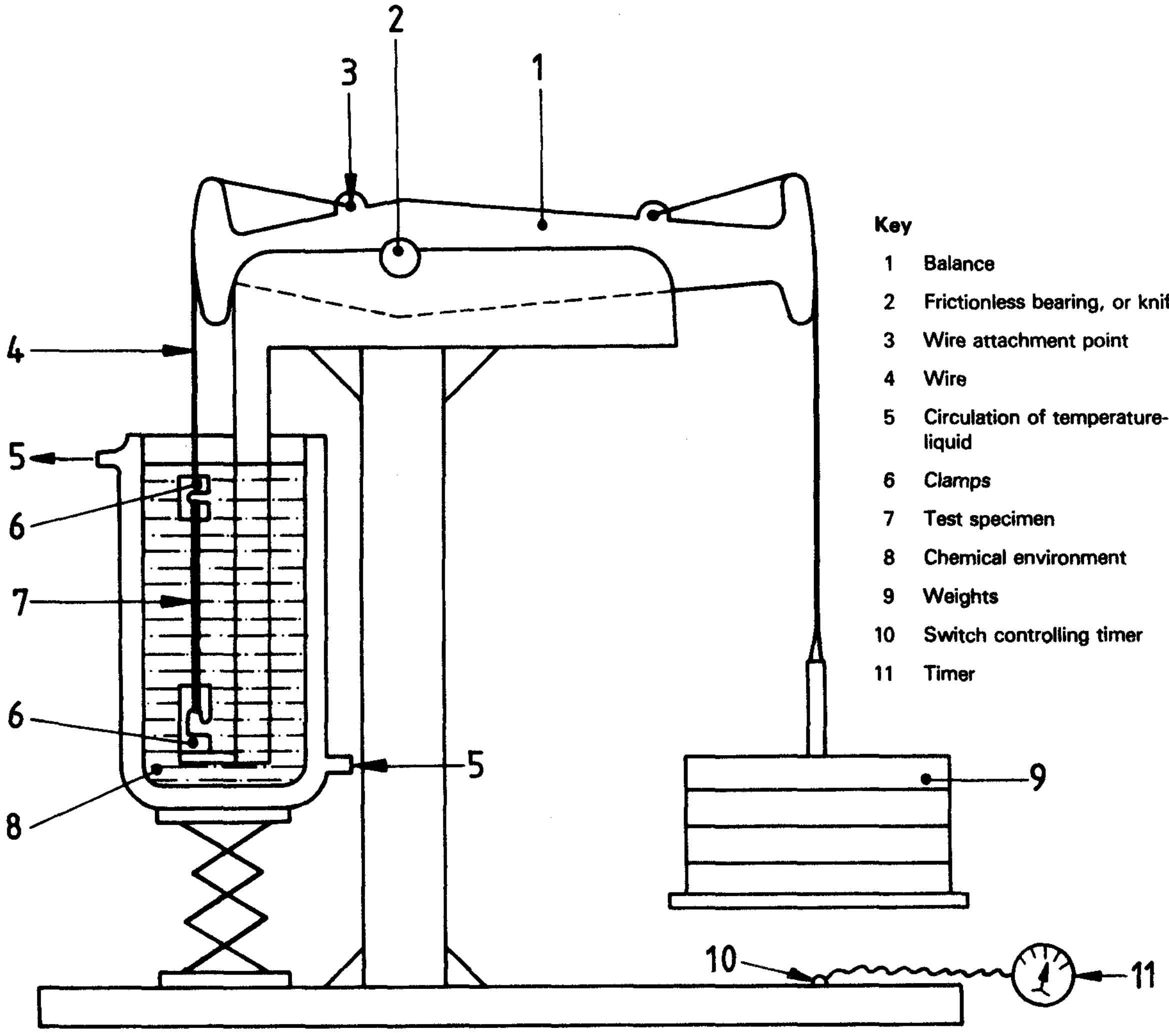
[1] This detergent is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.



Key

1	Cover support
2	Upper clamp
3	Lower clamp
4	Test specimen
5	Chemical environment
6	Glass container
7	Vibration damping base
8	Support
9	Microswitch connected to timer
10	Weights

Figure 1 — One type of apparatus for measuring fracture under constant stress



Key

1	Balance
2	Frictionless bearing, or knife edge
3	Wire attachment point
4	Wire
5	Circulation of temperature-controlled liquid
6	Clamps
7	Test specimen
8	Chemical environment
9	Weights
10	Switch controlling timer
11	Timer

Figure 2 — Balance type of apparatus with 2:1 amplification of load

7.3 Method B

Determine the time to rupture under a single stress as agreed between the interested parties, but not higher than the maximum permissible stress defined in 7.1.

7.4 Method C

Determine the times to rupture for a series of stresses. The loads shall be chosen in accordance with 9.7.

8 Test specimens

8.1 Shape and dimensions

Use the type 1 specimen specified in ISO/R 527, with all dimensions scaled down by a ratio of 1:2 as shown in figure 3.

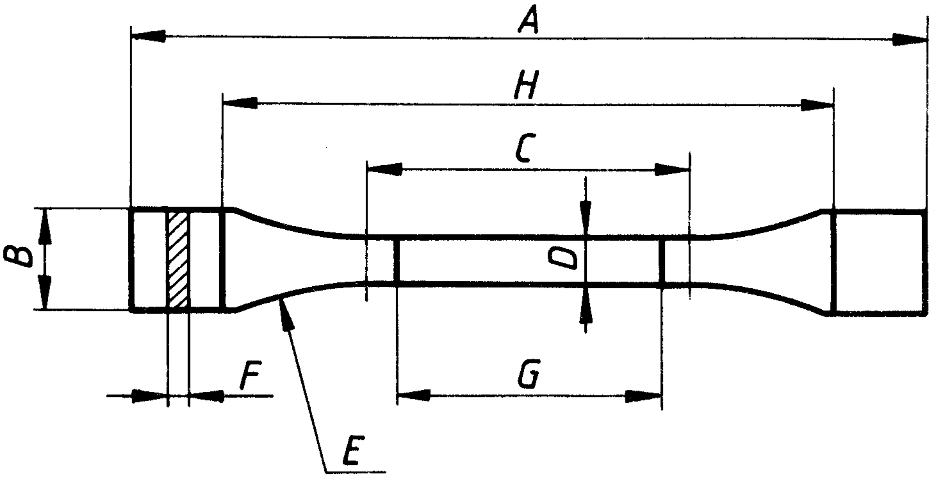
The preferred thickness is (2 ± 0,2) mm, but when the test specimens are prepared from products the thickness may be that of the product. Alternatively, a type 1 specimen 3 mm to 4 mm thick may be used.

Attention is drawn to the multipurpose specimen specified in ISO 3167.

8.2 Number

At least five specimens shall be tested at each tensile stress in the case of methods A and B and at least two specimens for each stress in the case of method C.

If the material is thought to be anisotropic, two sets of specimens shall be used, one set cut at right angles to the other in two of the principal directions.



Key

A	Overall length, minimum: 75 mm
B	Width at ends: 10 mm ± 0,5 mm
C	Length of narrow, parallel-sided portion: 30 mm ± 0,5 mm
D	Width of narrow, parallel-sided portion: 5 mm ± 0,5 mm
E	Radius, minimum: 30 mm
F	Thickness: 2 mm ± 0,2 mm
G	Distance between gauge marks: 25 mm
H	Initial distance between grips: 57 mm

Figure 3 — ISO/R 527 Type 1 specimen (scaled down 1:2)

8.3 Preparation

The specimens shall be prepared in accordance with the appropriate International Standard. If nothing is stated, specimens shall be machined from sheet or from products by the methods specified in ISO 2818.

If sheets are prepared from moulding materials, they shall be moulded in accordance with the relevant material specification or as agreed between the interested parties. Specimens shall not be cut with a die unless machining is impossible, for example with soft materials.

If specimens are prepared by moulding, the procedures shall be in accordance with ISO 293 or ISO 294.

NOTES

5 Environmental stress cracking of a specimen is influenced not only by the material, but also by the method of preparation. Materials should only be compared using specimens prepared in a similar manner and in the same state. Attention is drawn to ISO 2557-1 for the determination of level of shrinkage .

6 Injection-moulded test specimens often have a considerable amount of orientation. If the load is applied parallel to the direction of injection, the time to rupture may be significantly longer than in the transverse direction. If the specimens are anisotropic, it may be useful to carry out tests with the load applied in different directions relative to the direction of injection.

9 Procedure

9.1 Measure, to the nearest 0,01 mm, the thickness and the width of the central, parallel-sided portion of each specimen and calculate the force F, in newtons, to be applied, using the equation

$$F = \sigma \cdot A$$

where

σ is the stress, in megapascals, selected for the test (as described in 7.1);

A is the area of cross-section, in square millimetres, of the central, parallel-sided portion of the specimen.

NOTE 7 To determine the cross-sectional area it is recommended

a) to measure the thickness at each end of the parallel-sided portion and take the minimum value;

b) to measure the width of each face at each end of the parallel-sided portion and to take the mean value.

9.2 Heat the temperature-controlled bath or room (4.2) to the selected test temperature.

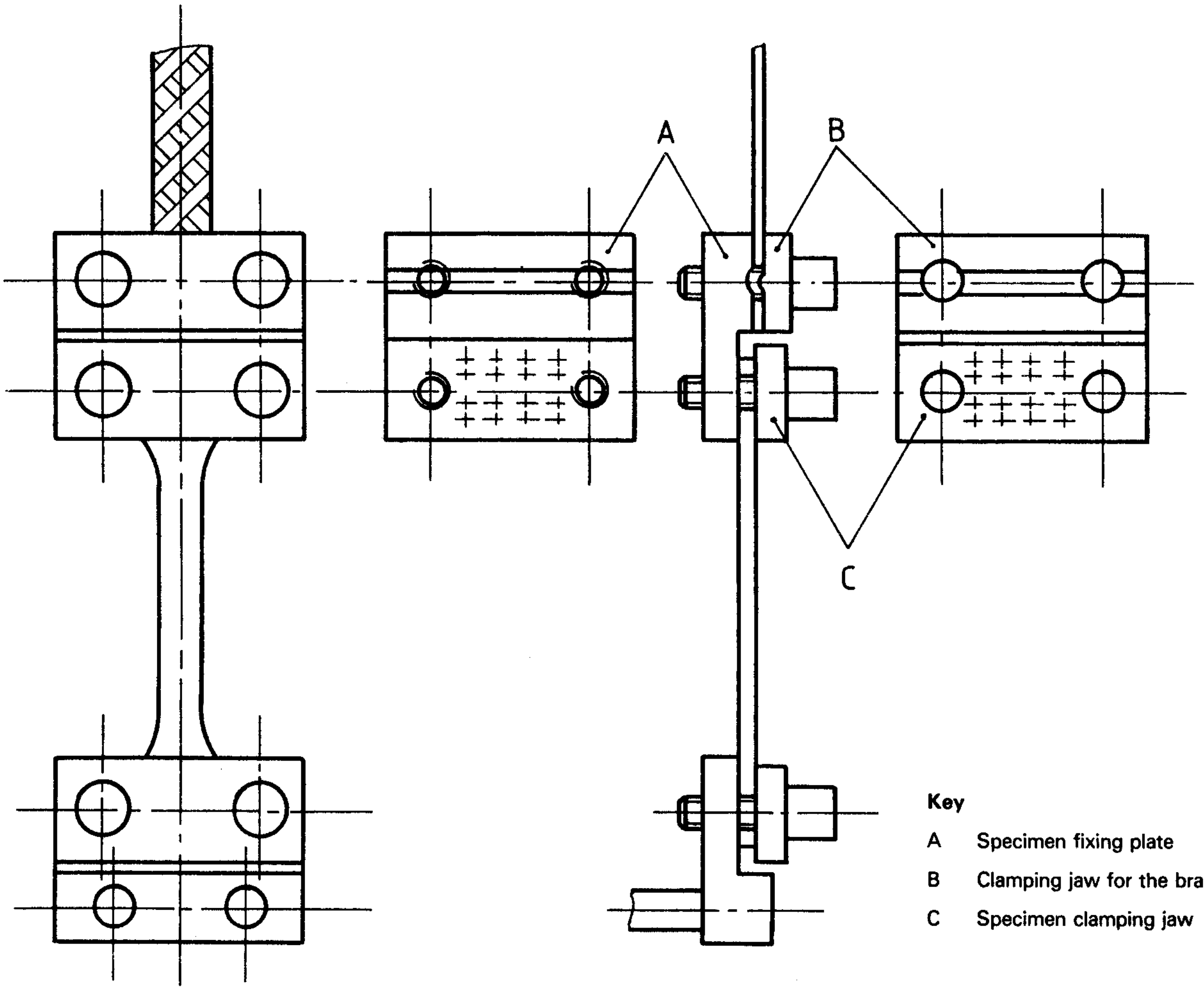
9.3 Insert the specimens in the clamps of the testing device (4.1) and immerse them in the test liquid or coat them with the chemical. Suitable clamp arrangements are shown schematically in figure 4.

9.4 After 15 min, apply the load F to each specimen, without shock, in such a way that the loading time is preferably between 3 s and 5 s and in any case less than 10 s. Start the timer (4.3) as soon as the load is applied ($t = 0$). Record the time to rupture for each specimen and the type of break (brittle or ductile).

If a liquid chemical environment is used, it shall be renewed with liquid from the same batch for each test specimen (apparatus with one station) or each group of test specimens (apparatus with several stations).

9.5 When method A is used, perform the test with a series of tensile stresses up to and including the maximum permissible stress as defined in 7.1.

NOTE 8 The 100 h stress is obtained by interpolation of a stress versus log time plot (see 10.1). If the logarithm of the arithmetic mean of the times to rupture is used, the longer times are overemphasized. A more conservative assessment of the times to rupture can be obtained by calculating the mean of the logarithms of the measured times to rupture, i.e. the geometric mean.



Key

A Specimen fixing plate

B Clamping jaw for the braid

C Specimen clamping jaw

Figure 4 — Example of suitable clamp arrangement

9.6 When method B is used, perform the test using a specified or agreed stress not higher than the maximum permissible stress as defined in 7.1 (see 7.3). If no break occurs after 1 000 h, terminate the test and record this fact in the test report.

9.7 When method C is used, perform the test using a series of stresses. The loads shall be chosen so as to fall within the range from 10 % to 90 % of the short-time tensile strength of the material and shall be selected from the following numbers: 1; 2; 3; 5; 7,5; 10 and their decimal multiples.

9.8 If required, carry out a parallel series of tests, as described in 9.5 or 9.6, in air or another reference environment.

10 Expression of results

10.1 Method A

Calculate the arithmetic mean and the standard deviation of the measured times to rupture. Plot the logarithm of the mean, in hours, as abscissa versus tensile stress, in megapascals, as ordinate and determine by interpolation the stress corresponding to a time to rupture of 100 h.

10.2 Method B

Calculate the arithmetic mean of the times to rupture, in hours, obtained from the five specimens and the standard deviation.

NOTE 9 For some purposes, the geometric, rather than the arithmetic, mean may be found useful because the logarithms of the times to rupture often show a better Gaussian distribution than the times to rupture.

10.3 Method C

Calculate the arithmetic mean of the times to rupture for each stress used. Plot the logarithm of each mean time to rupture in hours (as abscissae) versus tensile stress in megapascals (as ordinates).

11 Precision

The precision of these methods is not known because inter-laboratory data are not available in view of the variety of plastics materials and environmental conditions. These methods may not be suitable for use in the event of disputed results as long as no precision data are available.

12 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary to identify the material tested;

c) the chemical environment used;

d) the test temperature;

e) the number of specimens tested (if applicable, in each direction of anisotropy) and their width and thickness;

f) the procedure used for preparation of the specimens and, whenever possible, the elapsed time between their preparation and the beginning of testing;

g) the state of the specimens;

h) the conditioning duration and atmosphere;

i) the stresses applied;

j) the times to rupture, individual and mean values and standard deviations (methods A and B only), for each stress applied. If no rupture occurs after 1 000 h under the maximum permissible stress as defined in 7.1, report that fact;

k) for method A, the stress corresponding to 100 h time to rupture;

l) the type of break, i.e. brittle or ductile;

m) any operational details not specified in this International Standard, and any circumstance liable to have influenced the results;

n) results from parallel series of tests in air or another reference environment, if used.

Annex A
(informative)

Examples of stresses to be applied

Type of plastic	Temperature °C	Maximum stress to be applied MPa
Polyamide 66	55	30
Polycarbonate	55	40
Polycarbonate	23	50
PVC (unplasticized)	55	21
Polyethylene (high density)	55	4 to 7 depending on the molecular mass
Poly(methyl methacrylate)	55	25
Poly(methyl methacrylate)	23	40
Poly(oxymethylene)	55	28

NOTE 10 These values are given for information only. The maximum permissible stress is dependent upon the molecular mass of the polymer tested.

(Continued from second cover)

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 899-1 : 1993 Plastics — Determination of creep behaviour — Part 1 : Tensile creep	Nil	—
ISO 2557-1 : 1989 Plastics — Amorphous thermoplastics — Preparation of test specimens with a specified maximum reversion — Part 1 : Bars	Nil	—
ISO 2818 : 1994 Plastics — Preparation of test specimens by machining	IS 13360 (Part 2/Sec 4) : 1998 Plastic — Methods of testing : Part 2 Sampling and preparation of test specimens, Section 4 Preparation of test specimens by machining (*first revision*)	Identical
ISO 3167 : 1993 Plastics — Multipurpose test specimens	IS 13360 (Part 2/Sec 5) : 1997 Plastics — Methods of testing : Part 2 Sampling and preparation of test specimens, Section 5 Multipurpose test specimens	do
ISO 4599 : 1986 Plastics — Determination of resistance to environmental stress cracking (ESC) — Bent strip method	IS 13360 (Part 8/Sec 9) : 1997 Plastics — Methods of testing : Part 8 Permanence/chemical properties, Section 9 Determination of resistance to environmental stress cracking (ESC) — Bent strip method	do
ISO 4600 : 1992 Plastics — Determination of environmental stress cracking (ESC) — Ball or pin impression method	IS 13360 (Part 8/Sec 10) : 1998 Plastics — Methods of testing : Part 8 Permanence/chemical properties, Section 10 Determination of environmental stress cracking (ESC) — Ball or pin impression method	do

In the case of ISO 150, ISO 899-1 and ISO 2557-1, the Technical Committee responsible for the preparation of this standard has reviewed its contents and has decided that they are acceptable for use in conjunction with this standard.

For tropical countries like India, the standard temperature and the relative humidity shall be taken as 27 ± 2°C and 65 ± 5 percent respectively.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards : Monthly Additions'.

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Amendments Issued Since Publication

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